1. **Project Name:** **Characterization and Structural Modeling of Magnesia-Alumina Spinel Glass Tank Refractories**

2. **Lead Organization:** University of Missouri – Rolla
Department of Ceramic Engineering
222 McNutt Hall
Rolla, MO 65409

3. **Principal Investigator:** Dr. William L. Headrick
(573) 341-4188 (phone)
(573) 341-6934 (fax)
bill@umr.edu

4. **Project Partners:** Oak Ridge National Laboratory
(mechanical testing and thermophysical evaluation)
Dr. James G. Hemrick
Dr. Mattison K. Ferber
Dr. Hsin Wang

Saint-Gobain SEFPRO Refractories
(materials and technical support)

Glass Industry Advisory Committee – composed of representatives from glass and refractory industries (including AFG Industries Inc., PPG Industries Inc., Kimball, Saint-Gobain Containers, Thomson Multimedia Inc., Saint-Gobain SEFPRO, North American Refractories Co.)
(technical support)

5. **Date Project Initiated and FY of Effort:** October 1, 2001
Designed as a 1 year feasibility study on spinel refractory materials
Currently under a no-cost extension to complete additional creep and thermal conductivity testing.

6. **Expected Completion Date:** September 31, 2003

7. **Project Technical Milestones and Schedule:**

Project Objective: This project is intended to meet the need for characterization of key properties concerning a class of refractory materials (magnesia-alumina spinel based refractories) being considered as an alternative material to traditionally used silica).

Task Identification Number	Description	Planned Completion Date	Actual Completion Date	Comments
1	Initial Low Stress Creep Measurement	July 2002	September 2002	Results led to need for further work at higher stresses.
2	Corrosion Evaluation	March 2002	May 2002	
3	Thermal Conductivity Measurement	June 2002	On-going	Low temperature data has been collected, but higher temperature data is desired.
4	Microstructural Analysis	July 2002	July 2002	
5	Modeling	August 2002	On-going	Literature Review Complete. Model complete. New data from extension work will be incorporated into current model.
6	Additional High Stress Creep Measurement	September 2003	On-going	

8. **Current Project Milestones and Accomplishments:**

- Task 1: Creep testing has been completed on both bonded and fusion-cast spinel materials at low stresses and temperatures up to 1650^{o}C. Testing has shown substantial creep in the bonded material and very low levels of creep in the fusion-cast material. Lack of generated accumulated strain in the fusion-cast material has led planned work to further investigate the creep properties of this material at higher stress levels in hopes of generating significant creep rates.
- Task 2: Corrosion testing showed negligible deterioration of both the bonded and fusion-cast materials by high alkali containing solutions.
- Task 3: Thermal conductivity has been measured on both the bonded and fusion-cast material at temperatures up to temperatures of 1320^{o}C. Data is desired for these materials at higher temperatures and R&D is underway to modify the laser flash diffusivity technique to acquire such measurements. Measured thermal conductivity values have found to be on the order of those measured previously for fusion-cast alumina materials. These values are approximately five times higher than those measured for traditional silica refractories leading to concerns about heat loss through crowns composed of magnesia-alumina spinel materials.
- Task 4: Pre and post alkali exposure microstructures have been imaged. Images have been acquired using both optical microscopy and cathodoluminescence techniques. Microscopy shows the fusion-cast material to have a much smaller grain size and tighter grain structure than the bonded material which is believed to be the reason for the greater creep resistance shown by the fusion-cast material. Cathodoluminscence provides a chemical map of the microstructure and shows negligible change in microstructure and chemistry due to alkali exposure.
- Task 5: Models have been completed for silica, mullite and alumina crowns using previously collected data. Low stress data and thermal conductivity data collected to date has been used to develop a model for both fusion-cast and bonded spinel materials. When additional creep and thermal conductivity data are available, they will be incorporated into the current model.

- Task 6: The current creep frame at ORNL is being modified with a larger pneumatic cylinder and larger load cell to accommodate creep testing at higher stresses.

9. **Planned Future Milestones:**
During the remainder of the no-cost extension, creep will be measured at higher stresses to obtain more meaningful strain levels and thermal conductivity will be evaluated at higher temperatures. This is necessary to provide additional input data for the modeling effort. The newly acquired data will be used, in addition to the previously obtained lower stress data, in the completion of the modeling effort.

10. **Issues/Barriers:**
The fusion-cast spinel material shows extremely low amounts of creep at the low stresses originally proposed for testing (even at the extreme temperatures of 1650^{o}C). Therefore, the creep frame at ORNL is being modified to allow for testing at higher stresses in an effort to produce meaningful strain levels in this material. It was found that the spinel materials become transparent to the laser light at high temperatures, thus making the use of the laser flash diffusivity technique insufficient for high temperature measurements. R&D is underway to apply coatings to these materials that are stable at high temperatures and will allow for use of the laser flash technique at elevated temperatures.

11. **Intended Market and Commercialization Plans/Progress:**
The resulting database will be used in three ways. First, it will be made available to the glass and refractory manufacturers for assisting them in the selection of the appropriate refractory for glass tank furnaces. Second, the data will be used to refine existing models describing the stress, temperature and microstructural dependencies of the creep rate. Third, the data will be used in conjunction with finite element analysis (FEA) to predict the time-dependent deformation of a glass-tank crown. All three uses of the database will aid glass companies in estimating the current life of their glass tanks and to better plan future glass tank construction for more economical and energy efficient furnaces.

12. **Patents, publications, presentations:**

Project website: http://www.umr.edu/~bill/projects/creep/

American Ceramic Society Poster, May 2002

IMF Poster, July 2002

IMF Presentation, July 2002

Corhart Presentation, July 2002

Article to be published in August issue of Refractory Applications and News

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Dr. Hsin Wang (Oak Ridge National Laboratory)

Problem: There is a need for characterization of key properties concerning refractory materials for improved thermal efficiency and management in industrial combustion environments. Specifically, the glass industry desires thermomechanical property data on alternative refractory materials (to traditionally used silica) such as Magnesia-Alumina Spinel.

Results:

Microstructure

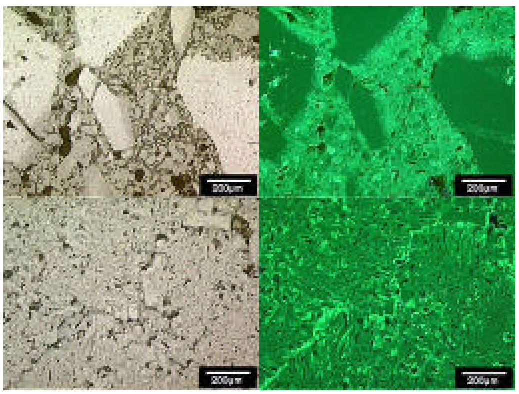

Figure 1: Optical Microscopy and Cathodoluminesce Microscopy of Bonded (Top) and Fusion-Cast (Bottom) Spinel

Thermal Conductivity

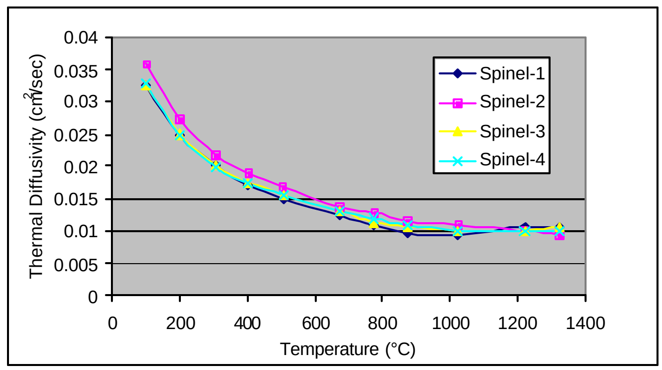

Figure 2: Characteristic Thermal Diffusivity of Spinel

Creep

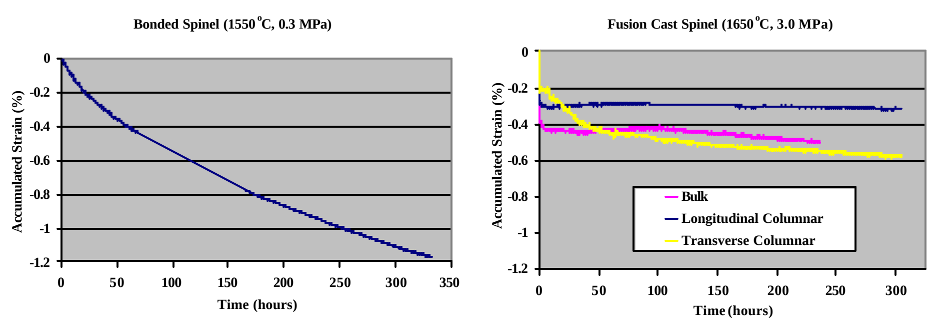

Figure 3: Accumulated Strain for Bonded and Fusion-Cast Spinel

Modeling

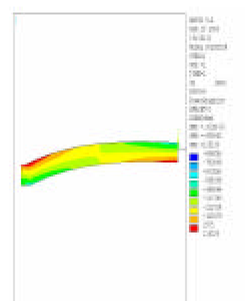

Figure 4: Sample FEA Crown Stress Model

Significance to Industries of the Future:

The resulting database information will be made available to the glass manufacturers for assisting in the selection of appropriate refractories for glass melting furnaces. Additionally, data will be used to refine existing models describing the stress, temperature and microstructural dependencies of the creep rate and data will be used in conjunction with finite element analysis (FEA) to predict the time-dependent deformation of glass-tank crowns. This will allow glass companies to estimate the current life of their glass tanks and to better plan future glass tank construction for more economical and energy efficient furnaces. If implemented, an energy savings opportunity of 90 trillion Btu/year could be addressed primarily through the enabling of oxy-fuel firing in reverberatroy glass melting furnaces.